MEASUREMENTS OF YOUNG’S MODULUS, POISSON’S RATIO, AND TENSILE STRENGTH OF POLYSILICON

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ABSTRACT

New techniques and procedures are described that enable one to measure the mechanical properties of polysilicon films that are 3.5 µm thick. Polysilicon is deposited onto a silicon substrate which is then etched away to leave a tensile specimen in the middle of the die. The grip ends of the structure are glued to the grips of a linear air bearing attached to a piezoelectrically actuated loading system. Strain is measured directly on the specimen with laser interferometry.

The specimens are fabricated at the Microelectronics Center of North Carolina with their MUMPs process. The results of 48 tests on five different sets of MUMPs specimens yield the following material properties: Young’s modulus = 169 ± 6.15 GPa, Poisson’s ratio = 0.22 ± 0.011, and tensile strength = 1.20 ± 0.15 GPa. These values have a reasonably low coefficient of variation which demonstrates the consistency of both the processing and the measurement techniques.

INTRODUCTION

Polysilicon is the most widely used structural material in current microdevices that are manufactured by surface micromachining. While its Young’s modulus has been measured with sufficient accuracy by various indirect methods, Poisson’s ratio and the tensile strength have not. These three mechanical properties (two related to stiffness and one related to failure) of a brittle material are required for intelligent design.

The mechanical testing of polysilicon is difficult because of the material is so thin. Three measurement issues arise: preparation and handling of specimens, friction in the loading mechanism, and strain measurement. The authors have developed techniques and procedures that permit the measurement of the Young’s modulus, Poisson’s ratio, and tensile strength of polysilicon film that is 3.5 micrometers thick. Moreover, this approach is similar in concept to the standardized tensile testing of large metal specimens in that a uniform stress is applied and strain is measured directly on the specimen.

Details of the material are presented in the first section, and then the unique specimen design and preparation are described. The key to these measurements is the ability to measure strain directly on the thin specimen; laser interferometry from gold lines deposited on the specimen surface is reviewed. The test machine, with its piezoelectric actuator and linear air bearing, is described. The results are presented and discussed in separate sections and then final conclusions are drawn.

MATERIAL

The polysilicon tensile specimens are manufactured at the Microelectronics Center of North Carolina (MCNC) and prepared for testing at the Applied Physics Laboratory of the Johns Hopkins University. MCNC’s DARPA-supported Multi-User MEMS Process (MUMPs) is typical of the processes commonly used to manufacture surface micromachined devices. Two layers of polycrystalline silicon are used to form the structural elements of the MEMS devices. The polysilicon layers are separated by sacrificial layers of phosphosilicate glass (PSG) and isolated from the supporting silicon substrate by a layer of silicon nitride. A final metal layer defines electrical contacts for the devices. When device fabrication is completed, the PSG layers are dissolved in an etching solution to free the mechanical structures.

The polysilicon films are deposited by low pressure chemical vapor deposition (LPCVD). The first polysilicon layer is 2.0 µm thick. It is deposited over a layer of PSG, and then covered with a second PSG film. The as-deposited silicon film is amorphous and must be annealed
at 1050°C for one hour to produce a fine grain, polycrystalline material with low internal stress. The annealing also dopes the polysilicon with phosphorus from the PSG layers above and below to make it conductive. Once the first polysilicon layer has been formed, the second 1.5 µm thick layer is deposited and annealed in an identical fashion. For the purposes of this investigation, the PSG that normally separates the two polysilicon layers was removed during processing. The second polysilicon layer was deposited directly on top of the first to produce a final combined thickness of 3.5 µm.

Figure 1 is a TEM photograph of the material through its thickness. The grains are generally columnar and perpendicular to the substrate; when viewed from the top, the grains are more circular in shape. The material is not completely isotropic; however, it is isotropic in the plane of the specimen.

The 3.5 µm thick by 0.6 mm wide tensile specimen is horizontal in the center of the figure and has large grip ends on either side. A rectangular area in the center of the die is etched away from the back to leave the tensile specimen stretched between the two grip ends as shown in Section A-A. The two grip ends are held together by two support strips as shown in the upper part of Figure 2. Gold lines are deposited onto the specimen to form a strain gage as described in the next section.

Release of the specimen begins by mounting the silicon die on a ceramic carrier using an adhesive wax between the front of the die and the carrier. After removing the polysilicon and oxide layers from the back of the die with a wet etch, the silicon nitride layer is patterned by photolithography. Reactive ion etching (RIE) is used to open a rectangular window in the nitride directly beneath the tensile portion of the specimen. With the silicon wafer exposed, the anisotropic etchant preferentially attacks the <100> crystal planes without significant lateral etching. The silicon nitride remaining on the back of the die serves as the etch mask while the nitride on the front of the die, between the substrate and the polysilicon specimen, provides an etch stop. Once the bulk silicon has been removed from the opening, the nitride layer on the front of the die beneath the specimen is removed by RIE. The oxide layer directly beneath the specimen is then removed in concentrated (49%) HF. Finally, the specimen is immersed in an organic solvent to dissolve the wax and free the die from the temporary carrier. A more complete description is given in [1].

After the specimen is released from the die, it remains fastened to the two grips which are held together by the two support strips. This structure is then mounted in the test machine by gluing down the grip ends. After the adhesive has cured, the two support strips are cut with a small diamond saw to free the tensile specimen. A specimen mounted in the grips and ready for testing is shown in Figure 3.

Figure 1. TEM photograph of the two-layer polysilicon specimen. the bottom layer is 2.0 µm thick and the darker top layer is 1.5 µm thick.

SPECIMENS

Polysilicon is deposited in the pattern shown in Figure 2 onto a one-centimeter square single crystal silicon die.

Figure 2. Schematic of the polysilicon pattern deposited onto a one-cm square die.

Figure 3. A polysilicon specimen mounted in the grips of the test machine with the support strips cut.
STRAIN MEASUREMENT

Strain is measured with the Interferometric Strain/Displacement Gage (ISDG) which is an optical technique for measuring the relative displacement between two reflective gage markers; see Figure 4. These markers are gold lines that are deposited on the specimen during manufacture as shown schematically in Figure 2. When the gold lines are illuminated with a laser, the diffracted reflections from each edge of a line overlap and interfere to produce fringes. As the gage markers move relative to each other, the fringe patterns also move; their motion can be measured with fringe detectors and related to the relative displacement change generated by strain (or displacement if the lines are across a crack).

\[
e = \frac{\lambda}{2d_0} \left( \frac{\Delta m_1}{\sin \alpha_1} + \frac{\Delta m_2}{\sin \alpha_2} \right)
\]

where \(\Delta m_1\) and \(\Delta m_2\) are the relative fringe shifts of patterns 1 and 2, \(d_0\) is the original gage length between the indentations, \(\lambda\) is the wavelength of the laser, and \(\alpha_1\) and \(\alpha_2\) are the angles between the incident laser beam and the fringe detectors.

The ISDG, as used for these tests, has a relative uncertainty of ± 3%. The original gage length can be measured to ± 0.5% and the relative uncertainty in locating minimums is ± 1%. The angle measurement contributes ± 1.5% to the uncertainty. With the current setup, the maximum sampling rate with real time calculations is ~ 13 data points per second. Applications of the ISDG over the past 25 years include measurements of biaxial strains at notch roots (three markers are placed in an orthogonal pattern), crack opening displacements of small cracks, dynamic strains and crack openings, creep strains, and strains/displacements at high temperatures. A 1982 review article [2] summarized the applications of the ISDG until that time, and a 1993 review [3] described various methods (including the ISDG) of measuring crack tip opening displacement. A NASA report [4] contains a detailed discussion of the optical principles and the practical aspects of an ISDG measurement system.

One constructs a biaxial ISDG by simply adding another pair of lines along with the necessary fringe detectors. Figure 5 is a photograph of such a biaxial strain gage. The lines are each 10 µm wide, 0.5 µm high and 200 µm long; the spacing is 250 µm. The four gold lines are the biaxial strain gage; it is interrogated by the laser interferometric system.

Figure 5. SEM photo of the biaxial strain gage. The lines are 250 µm apart.

TEST MACHINE

Figure 6 is a schematic of the test machine. The key feature is the linear air bearing that eliminates friction in the loading mechanism; it is attached to a 4.5 N load cell mounted on the end of a piezoelectric translator. Motion of the translator is controlled by a laboratory computer, and the test is run in displacement control.

The strain measurement system consists of a 10 mW He-Ne laser and two fringe detectors which are linear diode arrays - one for each pattern. The arrays have 512 diodes - each with an aperture of 13 µm by 2.5 mm - packaged in a 16-pin chip; the total size of the array is 2.5 mm by 12.6 mm. The arrays are inserted in circuit boards containing amplifying circuitry and mounted in plastic mini-boxes attached to translation stages which are mounted on adjustable rods. This provides the two degrees-of-freedom necessary to position the diode array in the center of the fringe pattern. Each array is accessed by a motherboard which samples each diode in turn and provides an output.
signal from 0 to 5 volts. The system shown in Figure 6 is for uniaxial strain measurement; biaxial strain measurement uses the same laser but requires two more fringe detectors.

RESULTS

A typical stress versus biaxial-strain result is plotted in Figure 7. Polysilicon is, as expected, a brittle material with a linear stress-strain curve. Note that a very slight preload is applied to the specimen before controlled loading is started. This is required to straighten the specimen in the grips (it buckles slightly in the vertical direction when the support strips are cut); this is a common procedure in mechanical testing. There are 353 data points in each curve plotted in Figure 7.

![BIAXIAL TEST # 18](image)

Figure 7. A typical stress biaxial-strain plot.

Table 1 presents the average results from five different MUMPs production runs. Ten specimens were tested from MUMPs 6, 10, and 11, and nine were tested from MUMPs 8 and 12. The biaxial strain measurements were initiated on specimens from MUMPs 11.

<table>
<thead>
<tr>
<th>MUMPs</th>
<th>Young’s Modulus (GPa)</th>
<th>Poisson’s Ratio</th>
<th>Tensile Strength (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>163 ± 4.78</td>
<td>-</td>
<td>1.23 ± .200</td>
</tr>
<tr>
<td>8</td>
<td>173 ± 6.94</td>
<td>-</td>
<td>1.27 ± .107</td>
</tr>
<tr>
<td>10</td>
<td>173 ± 2.00</td>
<td>-</td>
<td>1.14 ± .155</td>
</tr>
<tr>
<td>11</td>
<td>168 ± 7.15</td>
<td>0.22 ± .015</td>
<td>1.19 ± .155</td>
</tr>
<tr>
<td>12</td>
<td>168 ± 1.59</td>
<td>0.22 ± .003</td>
<td>1.17 ± .100</td>
</tr>
<tr>
<td>All Data</td>
<td>169 ± 6.15</td>
<td>0.22 ± .011</td>
<td>1.20 ± .150</td>
</tr>
</tbody>
</table>

There is no statistically significant difference at the 95% confidence level between the all-data tensile strength of 1.20 GPa and the average values for the different MUMPs runs.

The lowest modulus, 163 GPa for MUMPs 6, is statistically different from the average value of 169 GPa at the 95% confidence level. The modulus for MUMPs 10 is also, but that is because of its small standard deviation.

The coefficient of variation (CV = standard deviation / mean) for all data is 3.6% for the modulus. From our experience in testing large fine-grain steel specimens using standard techniques, the CV of the modulus can be expected to be less than 1/2 %. The strength CV for these tests is 12.5%, whereas one would expect CV = 5% for fine-grain steels. The CV for the Poisson’s ratio measurements is 5%. The scatter in the results is quite reasonable for these new measurements and indicates that both the test techniques and the manufacturing processes are consistent and reproducible.

DISCUSSION

There are other approaches to measuring Young’s modulus and strength of polysilicon and their test results are summarized in Table 2. However, we do not know of any other measurements of Poisson’s ratio for this material. The material properties in Table 2 are for polysilicon although the manufacturing processes, specimen shapes, and dopant levels vary.

<table>
<thead>
<tr>
<th>Reference</th>
<th>Year</th>
<th>Young’s Modulus (GPa)</th>
<th>Tensile Strength (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tabata [5]</td>
<td>1989</td>
<td>160</td>
<td>-</td>
</tr>
<tr>
<td>Tai [6]</td>
<td>1990</td>
<td>123</td>
<td>-</td>
</tr>
<tr>
<td>Walker [7]</td>
<td>1991</td>
<td>190</td>
<td>-</td>
</tr>
<tr>
<td>Koskinen [8]</td>
<td>1993</td>
<td>175 ± 25</td>
<td>2.7 - 3.4</td>
</tr>
<tr>
<td>Maier-Schn [9]</td>
<td>1995</td>
<td>162 ± 8</td>
<td>-</td>
</tr>
<tr>
<td>Michalicek [10]</td>
<td>1995</td>
<td>163</td>
<td>-</td>
</tr>
<tr>
<td>Biebl [12]</td>
<td>1995</td>
<td>168 ± 7</td>
<td>-</td>
</tr>
<tr>
<td>Biebl [13]</td>
<td>1995</td>
<td>170</td>
<td>-</td>
</tr>
<tr>
<td>Read [14]</td>
<td>1996</td>
<td>140 ± 20</td>
<td>0.695</td>
</tr>
<tr>
<td>Kahn [15]</td>
<td>1996</td>
<td>150 ± 30</td>
<td>-</td>
</tr>
<tr>
<td>Gupta [16]</td>
<td>1996</td>
<td>147 ± 10</td>
<td>-</td>
</tr>
<tr>
<td>Jones [17]</td>
<td>1996</td>
<td>-</td>
<td>1.87 ± 0.24</td>
</tr>
<tr>
<td>This work</td>
<td>1996</td>
<td>169 ± 6.2</td>
<td>1.20 ± 0.15</td>
</tr>
</tbody>
</table>

Tabata et al [5] used Newton interference fringes to measure the deflection of a pressurized 2 mm by 8 mm membrane of polysilicon. Tai and Muller [6] used a unique slider mechanism to measure Young’s modulus of heavily phosphorous-doped, unannealed polysilicon. Walker et al [7] tested square membranes of undoped polysilicon and found that Young’s modulus increased from 190 GPa for the control group to 240 GPa for specimens exposed to pure HF.
Koskinen et al [8] developed an extremely clever technique for testing long thin (1 µm by 3.5 µm) fibers of polysilicon that had various grain sizes. These were true tensile tests and the strain was inferred from crosshead motion. Maier-Schneider et al [9] used the membrane bulge test to measure Young’s modulus and found that annealed specimens had a higher value; that is the one reported in Table 2.

Michalicek et al [10] extracted their value of Young’s modulus from square flexure beam micromirrors by comparing the measured deflections with a sophisticated model. Their specimens were manufactured on the MUMPs 6 run and it is interesting that their value of 163 GPa is exactly the same as the average value reported in Table 2 for that production run.

Biebl and von Philipsborn [11] used a multiple beam structure to generate tensile forces in thin tensile specimens when the residual stress in the larger beams was released. This enabled measurement of tensile strengths. Biebl and colleagues used resonant structures [12] and cantilever beams [13] to obtain values of Young’s modulus for doped and annealed polysilicon that are very close to the average value reported for all data in this work.

Read and Marshall [14] tested small tensile specimens from MUMPs 8 and obtained modulus and strength values considerably smaller than reported here. Kahn et al [15] used a resonant structure to measure Young’s modulus and fracture toughness of boron-doped polysilicon. Gupta et al [16] used a cantilever beam and electrostatic pull-in arrangement to obtain the plate modulus $(E/(1 - \nu^2))$ of specimens from MUMPs 5 as 155 GPa. Assuming Poisson’s ratio to be 0.22, the Young’s modulus is then 147 GPa.

Jones et al [17] measured fracture strains in phosphorus-doped and annealed polysilicon with a cantilever beam structure having the beams bend in the plane of the film, not perpendicular to it as is the usual case. The effect of HF exposure time on the strength was studied; the strength value Table 2 is computed assuming $E = 170$ GPa and is for the minimal exposure.

The modulus values since 1993 in Table 2 seem to fall in two groups — those around 170 GPa and those around 150 GPa. The materials are not all different; the specimens tested here and those of Michalicek [10] are from MUMPs runs as are those tested by Read [14] and Gupta [16]. Aggregate theory can be used to compute both lower and upper bounds [18] on the Young’s modulus of a polycrystal from single crystal data. Those values are 164 GPa and 172 GPa respectively which enclose the results reported here.

There are fewer results on tensile strengths and considerable variation. The very small specimens of Koskinen [8] show the highest strength; the specimens tested in this work are much larger and show a lower strength. The concept that larger specimens of brittle materials have a higher probability of flaws is common in material science and needs to be investigated for polysilicon.

The membrane, beam, and similar tests take an inverse approach to obtaining Young’s modulus. They calculate the mechanical response (usually deflection) to a known applied external force, compare it to the measured response, and adjust to value of E to make a fit. This is fine if the boundary conditions are modeled exactly, but this can be difficult for a complex structure. That is the reason that the tensile test is the preferred and standardized method for measuring mechanical properties of materials; it is simple and straightforward.

CONCLUSIONS

Techniques and procedures have been developed to measure the mechanical properties of polysilicon. The difficult problems associated with specimen handling, friction during loading, and accurate strain measurement have been solved. This approach is suitable for other thin films; one would need to develop the techniques for depositing and releasing the tensile specimen.

Tensile tests are the standard method for determining the mechanical properties of materials whether they ARE linear and brittle like polysilicon or nonlinear and ductile like nickel. The imposition of a uniform stress field eliminates the effect of stress gradients which occur in specimens loaded in bending. One can then determine a true material property, not a mechanical property that is dependent on the shape of the specimen. From that point of view, this approach has advantages over bulge and beam tests.

There is sufficient evidence now to establish a nominal value of Young’s modulus for LPCVD polysilicon; that value is $170 \text{ GPa} \pm 4\%$. That is based on the nearly 50 tests of this work as well as other measurements on similar materials. Modulus values will be different for polysilicon that is produced and processed differently, but the values should be similar.

There is no other evidence for the Poisson’s ratio value of 0.22 for polysilicon. However, Poisson’s ratio for silicate glasses is approximately 0.20 - 0.25 [19] so the value seems reasonable.

One cannot be quite so certain about the tensile strength. Although the scatter in the results reported here is acceptable, there may well be a size effect on the breaking strength. This is an area for continuing research.
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